Low Hydrogen Embrittlement Alkaline Zinc-Nickel Plating for High Strength Steels

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for
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LHE Cadmium Plating

- Used Extensively on High Strength Steel Aerospace Parts
  - Process is Non-Embrittling
    - Uses a Modified Bright Cadmium Plating Process
    - Plate at High Current Density, No Brighteners and Higher NaCN/CdO Ratio to form a Dull (Porous) Plating
      - Hydrogen Baked Out at 375^{o} F for 24 Hours
        - » Verified by ASTM F 519 Sustained Load Test or ASTM F 326 Electronic Measurement
  - HS Steel with LHE Cadmium Appears to Have Good Resistance to Hydrogen Re-Embrittlement
    - No Concerns for Hydrogen Embrittlement When Exposed to Maintenance Fluids
      - Water and Salt Water Exposure Can Be Challenging

Zinc-Nickel Plating

- Zn-Ni Plating Appears to be Good Replacement for LHE Cadmium
  - Boeing Acid Zn-Ni Process is Non-Embrittling
    - Passes ASTM F 519 Sustained Load Tests - BUT
      - Plating Process is Not Operator Friendly
      - ASTM F 346 Electronic Measurement (or Similar Method) Cannot Be Used
  - Previously Tested Alkaline Zn-Ni Processes Were Sometimes Embrittling
    - Plating Process is Operator Friendly - BUT
      - Needed a Nickel Strike to Pass ASTM F 519 on a Consistent Basis

LHE Alkaline Zn-Ni Plating

- C-17 Pollution Prevention Project Started in October 2003
  - Develop an LHE Version of Alkaline Zn-Ni Plating
    - Look at Different Zn-Ni Formulas with Nickel Composition of 5 to 15%
    - Remove Brighteners and Other Additives to Make Plating Dull (Porous)
    - Vary the Current Density

LHE Alkaline Zn-Ni Plating

- Boeing Teamed with Dipsol of America to Develop an LHE Alkaline Zn-Ni Plating
  - Dipsol Produces IZ-260 Alkaline Zn-Ni Plating
    - Used by Several DoD and Aerospace Subcontractors
      - IZ-260 Has 5 to 8% Nickel – Balance Zinc
    - Dedicated R&D Laboratory in Tokyo, Japan
    - Excellent Technical Support at Laboratory in Livonia, MI
    - Dipsol Understands Zn-Ni Plating Chemistry

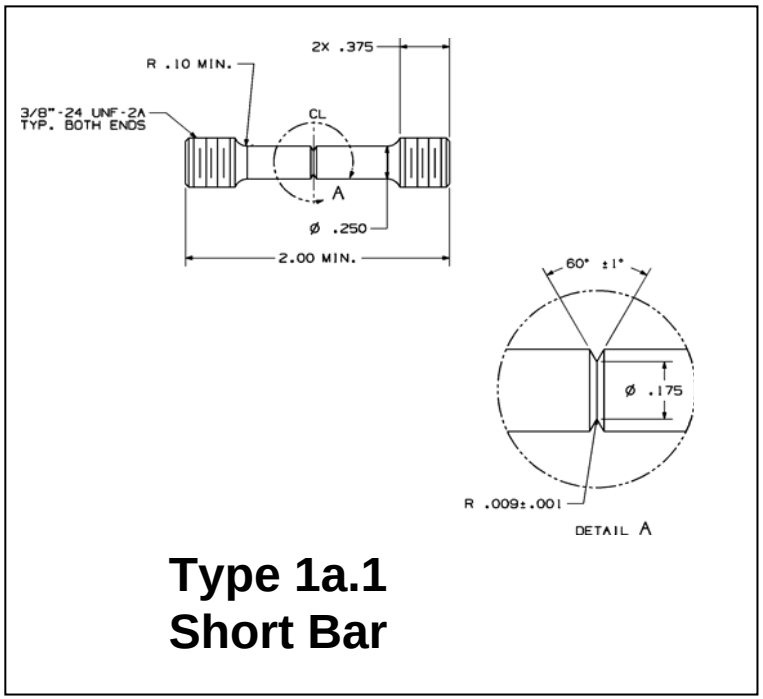
Phase I Testing

- Evaluate 7 LHE Alkaline Zn-Ni Formulas Proposed by Dipsol
  - #1 – Standard IZ-260 with Brighteners
  - #2 – Standard IZ-260 with No Brighteners
  - #3 – Aged IZ-260 Solution
  - #4 – IZ-260 with Double IZ-B Additive
  - #5 – Standard IZ-260 Plated at Low Current Density
  - #6 – IZ-260 Applied Over Nickel Strike
  - #7 – High Nickel Content Version of IZ-260 That Contains 12 to 15% Nickel

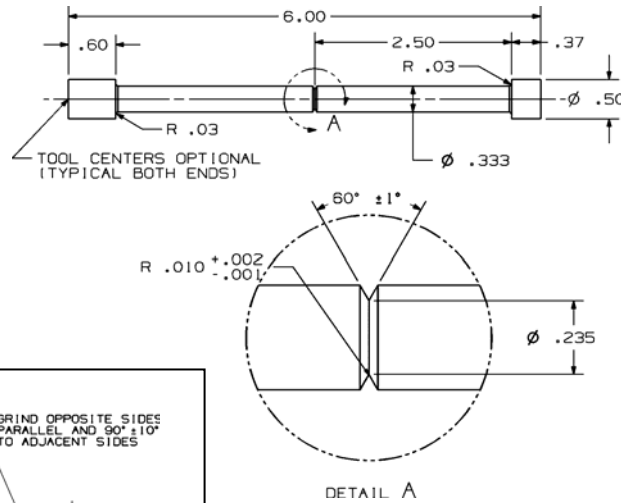
Phase I – Screening Tests

- Looked at 7 LHE Alkaline Zn-Ni Formulas
  - Conducted 3 Sets of Hydrogen Embrittlement Tests
    - Type 1a.1 With Acid Etch Prior to Plating
    - Type 1a.2 and 1e With Acid Etch Prior to Plating and Baked or Not Baked After Plating
    - Type 1a.2 With No Acid Etch Prior to Plating
  - Perform Adhesion, Thickness and Corrosion Testing
  - Down Select to One LHE Alkaline Zn-Ni Formula for Phase II Testing

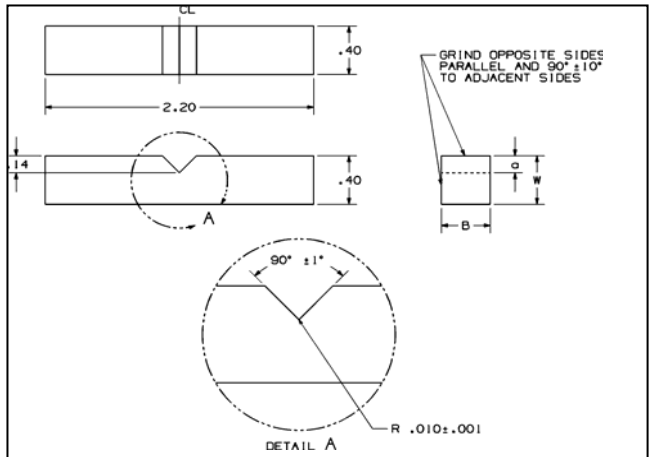
Hydrogen Embrittlement Test Specimens



Type 1a.1
Short Bar



Type 1a.2
Long Bar



Type 1e – ISL Square Bar

Test Specimens Per ASTM F 519-05

Phase I - HE Test Results

	Test #1	Test #2	Test #2	Test #2	Test #3
LHE Zn-Ni Formula*	**Type 1a.1 - SL**	**Type 1a.2 - SL**	**Type 1e - RSL**	**Type 1e - SL**	**Type 1a.2 - SL**
	Acid Clean	**Acid Clean**	**Acid Clean**	**Acid Clean**	**No Acid Clean**
1	**P,P,P**	**F,F,F**	**P**	**F**	**F,F,F**
2	**P,P,P**	**F,F,F**	**P**	**P**	**F,F,F**
2 - NO BAKE	NO TEST	NO TEST	**P**	**F**	NO TEST
3	**P,P,P**	**F,F,F**	**P**	**F**	**P,P,P**
4	**P,P,P**	**F,F,F**	**P**	**P**	**P,P,P**
4 - NO BAKE	NO TEST	NO TEST	**P**	**F**	NO TEST
5	**F,F,P**	**F,F,F**	**P**	**P**	**F,F,F**
6	**P,P,P**	**P,P,P**	**P**	**P**	**P,P,P**
6 - NO BAKE	NO TEST	NO TEST	**P**	**F**	NO TEST
7	**P,P,P**	**F,P,P**	**P**	**P**	**P,P,P**
7 - NO BAKE	NO TEST	**F,F,F**	**F**	**F**	NO TEST

Notes:

RSL = Rising Step Load (20/5/1 = 20 steps at 5% NFS per step and hold for 1 hour at each step
SL = Sustained Test Load (Hold at 75% NFS for 200 hours)
Type 1a.2 and 1e plated 100%, Type 1a.1 had no plating on threaded ends

*** LHE Zn-Ni Formulas**
1 - IZ 260 Standard Conditions (with 0.5% IZ 260S)
2 - IZ 260 without IZ 260S
3 - IZ 260 Aged Bath
4 - IZ 260 with double IZ-B
5 - IZ 260 at 1 A/dm^2 (low current density)
6 - IZ 260 over Nickel Strike
7 - IZ 260 with High Nickel Content

Phase I – Adhesion & Thickness

Specimen Type	1x4	1x4	1x4	1x4	4x6 - Unscribed	4x6 - Scribed	1x4
Formula # and Plating Type	% Ni by XRF[1]	Thickness by XRF[1] (mils)	Thickness by Mag Induction[2] (mils)	Thickness by Metallography[3] (mils)	Thickness by Mag Induction[2] (mils)	Thickness by Mag Induction[2] (mils)	Adhesion Test[4] Result
#1 - IZ-260 with 0.5% IZ-260S	8	0.49	0.35 - 0.39	0.27 - 0.32	0.5 - 0.7	0.44 - 0.50	Pass
#2 - IZ-260 with 0% IZ-260S	8.2	0.48	0.47 - 0.53	0.40 - 0.52	0.58 - 0.64	0.48 - 0.62	Pass
#3 - IZ-260 - Used / Aged Bath	7.3	0.61	0.29 - 0.33	0.29 - 0.31	0.31 - 0.43	0.40 - 0.46	Pass
#4 - IZ-260 with double IZ-B	6.4	0.50	0.36 - 0.40	0.37 - 0.40	0.44 - 0.56	0.43 - 0.57	Pass
#5 - IZ-260 plated at low current	7.3	0.82	0.52 - 0.56	0.42 - 0.48	0.47 - 0.65	0.46 - 0.54	Pass
#6 - IZ-260 with Nickel Strike	9	0.78	0.39 - 0.45	0.45 - 0.47	0.52 - 0.62	0.46 - 0.56	Pass
#7 - IZ-260 with High Ni Content	15.6	0.69	0.51 - 0.55	0.49 - 0.53	0.48 - 0.66	0.49 - 0.57	Pass
#8 - Cadmium (control)	-	-	-	-	-	0.25 - 0.31	-

Notes:

1 - %Ni composition and thickness of zinc-nickel determined by x-ray fluorescence method at Dipsol of America.

2 - Thickness measured with a DeFelsko gage using magnetic induction.

3 - Thickness measured with a metallographic cross-section of the zinc-nickel plating.

4 - Adhesion measured by doing a bend-to-break on 1x4 test strips.

Phase I – Corrosion Test

#1 thru 7 are LHE Alkaline Zn-Ni Formulas

#8 - Cadmium

ASTM B 117 – 816 Hours Exposure

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Phase II Testing

- Based on Phase I Test Results
  - Down Select to Formula #7 for Phase II Testing
    - Identified as LHE Zinc-Nickel Plus
- Phase II HE Testing
  - Hydrogen Embrittlement and Re-Embrittlement Testing with 1a.1 and 2a
    - Re-Embrittlement Test Specimens Exposed to Distilled Water and 3.5% Salt Water

Phase II – HE Test Results

Test Description	Specimen Type	ID No.	200 Hour Result (Pass/Fail)	ISL After 200 Hour Test (% NFS)
Set #1 - Plate Entire Specimen	1a.1	AQ3789	Pass	80
Set #1 - Plate Entire Specimen	1a.1	AQ5767	Pass	85
Set #1 - Plate Entire Specimen	1a.1	AQ3623	Pass	90
Set #1 - Plate Entire Specimen	1a.1	AQ3675	Pass	80
Set #2 - Plate at 3 Times Current Density	1a.1	AS1279	Pass	80
Set #2 - Plate at 3 Times Current Density	1a.1	AS1487	Pass	90
Set #2 - Plate at 3 Times Current Density	1a.1	AS1026	Pass	85
Set #2 - Plate at 3 Times Current Density	1a.1	AS1248	Pass	85
Set #3 - Plate with No Preplate Acid Activation	1a.1	AS1385	Pass	95
Set #3 - Plate with No Preplate Acid Activation	1a.1	AS1085	Pass	90
Set #3 - Plate with No Preplate Acid Activation	1a.1	AS1040	Pass	90
Set #3 - Plate with No Preplate Acid Activation	1a.1	AS1281	Pass	95
Set #4 - Plate with Preplate Acid Activation	1a.1	AS1264	Pass	90
Set #4 - Plate with Preplate Acid Activation	1a.1	AS1198	Pass	90
Set #4 - Plate with Preplate Acid Activation	1a.1	AS1421	Pass	90
Set #4 - Plate with Preplate Acid Activation	1a.1	AS1148	Pass	85
Set #5 - Plate with Preplate Acid Activation	2a	44911-12	Pass	-
Set #5 - Plate with Preplate Acid Activation	2a	44911-47	Pass	-
Set #5 - Plate with Preplate Acid Activation	2a	44911-54	Pass	-
Set #5 - Plate with Preplate Acid Activation	2a	44911-1	Pass	-

Hydrogen Embrittlement Results for LHE Zinc-Nickel Plus

ENVIRONMENTAL ASSURANCE

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Phase II – Re-Embrittlement Tests

Re-Embrittlement Test Fluid	Specimen Type	ID No.	150 Hour Result (Pass/Fail)
Distilled Water	1a.1	AS1224	Pass
Distilled Water	1a.1	AS1166	Pass
Distilled Water	1a.1	AS1368	Pass
Distilled Water	1a.1	AS1169	Pass
3.5% Salt (NaCl) Water	1a.1	AS1001	Pass
3.5% Salt (NaCl) Water	1a.1	AS1415	Pass
3.5% Salt (NaCl) Water	1a.1	AS1328	Pass
3.5% Salt (NaCl) Water	1a.1	AS1286	Pass
Distilled Water	2a	44911-42	Pass
Distilled Water	2a	44911-134	Pass
Distilled Water	2a	44911-41	Pass
Distilled Water	2a	44911-76	Pass
3.5% Salt (NaCl) Water	2a	44911-42	Pass*
3.5% Salt (NaCl) Water	2a	44911-134	Pass*
3.5% Salt (NaCl) Water	2a	44911-41	Pass*
3.5% Salt (NaCl) Water	2a	44911-76	Pass*

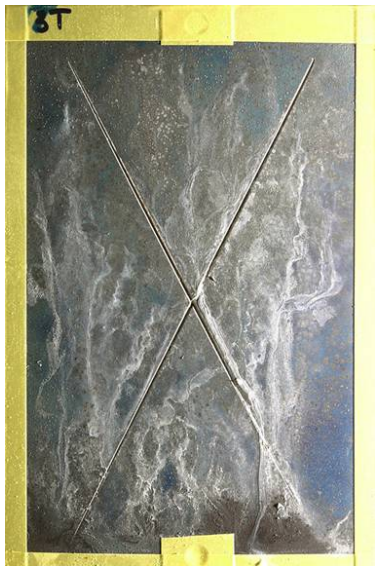
* 2a Test Specimens that passed the 150 hour distilled water test were used for the salt water test.

Hydrogen Re-Embrittlement Results for LHE Zinc-Nickel Plus

Phase II – Corrosion Tests

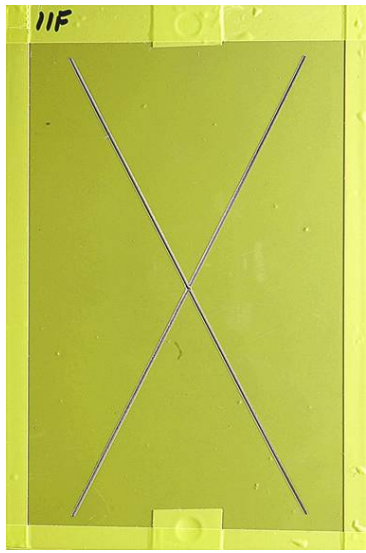
Bare Scribed LHE Zn-Ni Plus

0 Hours

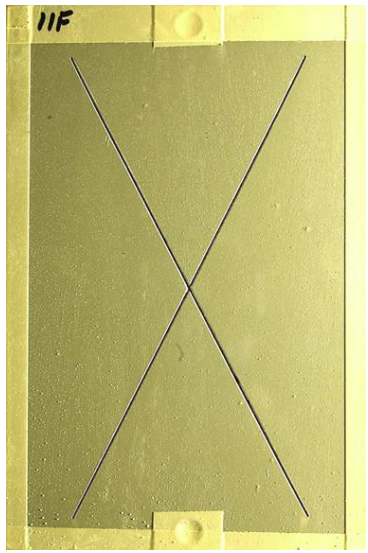


500 Hours

Painted* Scribed LHE Zn-Ni Plus



0 Hours



500 Hours

*** MIL-PRF-23377**

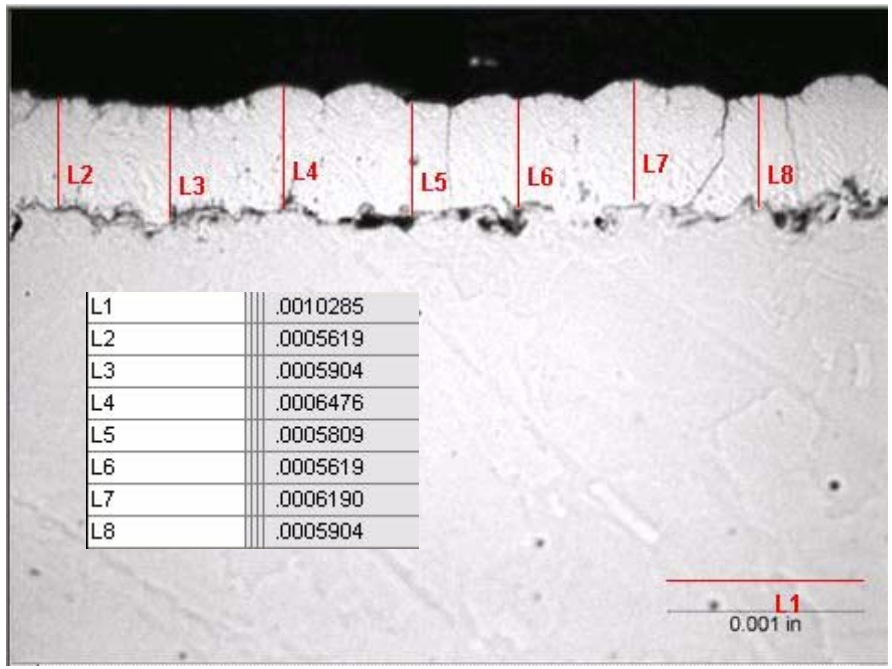
ASTM B117 Salt Spray Test Results

Phase II – Throwing Power

- Hull Cell Using 7.5 x 20 cm Test Panel
  - Zn-Ni Plated Along Entire Length of Panel
    - High Current Density End (14.8 A/dm^2)
      - 0.6 mils and 15.9 % Ni
    - Low Current Density End (0.3 A/dm^2)
      - 0.02 mils at 11.3 % Ni
- 60^o Bent Cathode
  - Used 7.5 x 20 cm Hull Cell Test Panel
    - High Current Density End (0.5 cm from open end)
      - 1 to 1.2 mils and 17.2 % Ni
    - Low Current Density End (9.5 cm from open end)
      - 0.1 mils and 14.7 % Ni

Phase II – Thickness and Adhesion

- LHE Zn-Ni Plus Has Good Adhesion
  - Passes Bend-To-Break Tests
- Thickness Control is Very Good



Next Steps

- Install Plating Tank at Boeing – St. Louis with Dipsol LHE Zinc-Nickel Plus
  - Perform More Hydrogen Embrittlement Tests
  - Perform Fatigue Tests
  - Perform Lubricity Tests
  - Optimize Operating Parameters
    - Verify Operating Limits of Plating Bath
  - Plate Parts with Complex Geometries
    - Determine Need for Auxiliary Anodes and Special Tooling
      - Plate ID of Tubular Parts
- Qualify LHE Zinc-Nickel Plus for C-17 Program
  - Create DPS for LHE Zinc-Nickel Plus
    - Identify Process Controls
    - Hydrogen Embrittlement Test Methods
    - Select Repair Procedures